

## Synthesis, characterization and crystal structure of a dodeca-nuclear cobalt (II)cube-octahedron

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**Abstract:** The new cobalt (II) complex,  $[\text{Co}_{12}(\text{cpip})_{12}(\text{H}_2\text{O})_{12}]\cdot 18.56\text{H}_2\text{O}$  where  $\text{H}_2\text{cpip} = 2\text{-}(\text{carboxy-phenyl})\text{-amino-propanoic acid}$ , was isolated as pink-like prism crystals from the reaction of  $\text{Co}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$  and  $\text{H}_2\text{cpip}$  in an ethanolic solution. The complex was characterized by Fourier Transform Infrared Spectroscopy (FTIR) and X-ray single-crystal diffraction. The symmetric unit of the title complex contains twelve cobalt atoms arranged in a cube-octahedron. Each divalent cation of cobalt is iso-octahedrally coordinated to four oxygen atoms of two cpip2- ligands in the equatorial plane. The axial plane is occupied by an amino nitrogen atom of one of the cpip2- ligands and an oxygen atom of a water molecule. The lattice water molecule in the metallocycle cavity involved OH---O hydrogen bonding with the coordinated water molecules.

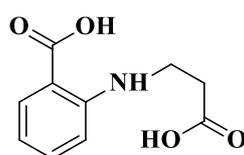
**Keywords:** Cube-octahedron; Crystal structure; Dodecahedron; Octahedral; Hydrogen bonding.

### 1. Introduction

The design, synthesis, and study of highly nucleated metallocyclic complexes have become the most active area of coordination chemistry<sup>1,2,3</sup>. Polynuclear metal complexes are hybrid metal-organic compounds where the organic or inorganic ligand holds the metal ions<sup>4</sup>. In particular, polynuclear metal compounds often exhibit unique structural features and combine physical properties such as magnetism and luminescence for drug discovery and catalyzing new product development<sup>5</sup>. Transition metals such as  $\text{Co}^{2+}$ ,  $\text{Mn}^{3+}$  and  $\text{Fe}^{3+}$  with polynuclear homometallic and heterometallic are good candidates for constructing single magnetic molecules (SMMs)<sup>6</sup>. Much work has been focused on complexes containing metal ions with unquenched orbital angular momentum, especially those containing high-spin Co (II) ions, because of their significant magnetic anisotropy.

It may also have potential application in information storage, and quantum tunnel magnetization<sup>7,8</sup>. Only a few Co (II) clusters have been investigated, which

may be ascribed to the absence of a facile synthesis route that prevents the Co (II) from oxidation in the air to Co (III)<sup>9</sup>. Single-molecule magnet in cobalt (II) clusters are much less known than it is in manganese (II) / (III) / (IV) clusters, such as  $[\text{Mn}_{12}\text{O}_{12}(\text{CH}_3\text{COO})_{15}(\text{CH}_3\text{CN})]^{+}$ . So, polynuclear of 3d-metal ions such as Co(II) promises a bright future due to their large magnetic anisotropy<sup>10</sup>. We have been interested in the design of ligands that favor the formation of polynuclear species and may control the structure. Polydentate ligands with carboxylate groups exhibit vibrant coordination chemistry due to their ability to act as terminal monodentate, chelating a metal center. To generate polynuclear complexes, they can also act as a bridge between transition metal ions in different modes, such as syn-syn, syn-anti, and anti-anti. At the same time as they can provide an efficient means to transmit magnetic information between adjacent metal ions.<sup>11</sup> The polycarboxylate ligand  $\text{H}_2\text{cpip}$  shown in (Scheme 1) was employed to synthesize the title complex. In this paper, we present the synthesis and structure of the title complex.



Scheme 1

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## 2. Experimental section

### 2.1. Materials and methods

All chemicals and solvents used in this work were obtained commercially from Aldrich or BDH and used without further purification. All manipulations were carried out under atmospheric pressure. Elemental analysis was performed on a Vario EL (III) elemental analyzer (C, H, and N) (Bruker Analytik GmbH, Hanau, Germany). FTIR spectra were recorded at room temperature with a Bruker IFS-250UPS/IR (Bruker Analytik GmbH, Hanau, Germany) over 400- 4000  $\text{cm}^{-1}$  range with a resolution of 4  $\text{cm}^{-1}$ .

### 2.2. Synthesis

#### 2.2.1 2-(carboxyphenyl) aminopropionic acid ( $\text{H}_2\text{cpip}$ )

This ligand was prepared using the previously described method<sup>12</sup>. A 5N NaOH (15ml, 0.073mol) was added with stirring to a solution of 2-aminobenzoic acid (10g, 0.073 mol) in 10ml of water. After dissolving the 2-aminobenzoic acid, chloropropanoic acid (26g, 0.225 mol) was added, and the solution was heated to 60°C in an oil bath. A 5N NaOH aqueous solution was added dropwise to the stirred solution (45ml). After heating for 15 hours, the temperature was lowered to room temperature. The volume of the solution was reduced, and (0.50 mol) of concentrated HCl was added to produce a brown precipitate of the pure product with a 75% yield. Found: 57.30; H, 5.28; N, 6.72; Calc.: C,57.42; H, 5.26; N, 6.7%. IR ( $\text{cm}^{-1}$ ) 3366 m, 3083 w, 1704 vs, 1650 s, 1575 s, 1480 m, 1439 s, 1320 m, 1240 s, 1106 m, 907 w, 747 s.  $^1\text{H-NMR}$  (DMSO- $d_6$ ):  $\delta$ 2.5 (tr, 2H),  $\delta$ 3.5 (tr, 2H),  $\delta$ 7.35 (m, 4H).

#### 2.2.2 Synthesis of $[\text{Co}_{12}(\text{cpip})_{12}(\text{H}_2\text{O})_{12}] \cdot 18.56 \text{H}_2\text{O}$ (1)

The title complex (1) was prepared by the reaction of  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (0.130 g, 0.51mmol) and the ligand  $\text{H}_2\text{cpip}$  (0.068 g, 0.26 mmol) with triethylamine in about 20ml of EtOH with heating for 50 minutes gave the pink solution and a pink-red precipitate. The solution was filtered off, and the filtrate was left undisturbed for one week. Pink-like prism crystals of complex (1) were slowly formed. FTIR and single-crystal X-ray diffraction characterized the final product. Selected IR data ( $\text{cm}^{-1}$ ): 3440 br, 3366 s, 1600 vs, 1560 s, 1500 sh, 1390 sh, 1220 m, 1250 m, 1100 m, 810 m, 615 w.

#### 2.2.3 Details of crystal structure determination and refinement of 1

The title complex's suitable pink-like prism single crystals were obtained by slow solvent evaporation at ambient temperature. A crystal of 0.205 x 0.188 x 0.072mm was selected for data collection. Data were collected on Bruker–Nonius Kappa CCD

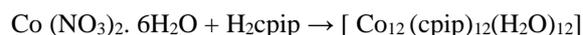
diffractometer at 293(2)K using graphite monochromator Mo- $\text{k}\alpha$  radiation ( $\lambda=0.71073\text{\AA}$ ). The structure was solved by direct methods using SHELXT<sup>13</sup> and refined using total matrix least-squares based on F2 using SHELXL-2014<sup>14</sup>. Non-hydrogen atoms were refined anisotropically. All hydrogen atoms were located by difference Fourier synthesis and refined isotropically<sup>15</sup>. Further details regarding the data collection, crystal, and final refinement parameters are found in (Table1).

Additional material from the Cambridge Crystallographic Data Center comprises thermal parameters and remaining bond distances and angles (CCDC No. 1966433). These data can be obtained free of charge from The Cambridge Crystallographic Data Center (CCDC), 12 Union Road, Cambridge CB2 IEZ, UK (fax +44(0)1223-336033; e-mail: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk)).

## 3. Results and discussion

### 3.1. Crystal structure of the title complex (1)

An interesting metallocycle cobalt (II) complex  $[\text{Co}_{12}(\text{cpip})_{12}(\text{H}_2\text{O})_{12}]$  was isolated as pink-like prism crystals from the reaction of  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  and the ligand  $\text{H}_2\text{cpip}$  in an ethanolic solution.

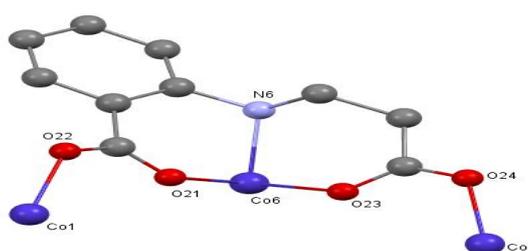


The IR spectrum of the title complex exhibits a strong absorption band at 1600  $\text{cm}^{-1}$  assignable to the carbonyl of the carboxylate group. About 100  $\text{cm}^{-1}$  shifted this band to a lower frequency relative to the corresponding frequency of the free ligand (1704  $\text{cm}^{-1}$ ), and another two bands as shoulders at 1500 and 1390  $\text{cm}^{-1}$  indicate the asymmetric and symmetric stretching frequencies of the carboxylate groups. The slight difference between  $\nu_{\text{as}}$  (COO) and  $\nu_{\text{s}}$  (COO) is characteristic of bidentate coordination. The absorption band of the amine group of the free ligand  $\nu(\text{NH})$  at 3366  $\text{cm}^{-1}$  disappeared, suggesting the involvement of the amine group in the coordination. The  $\nu(\text{OH})$  absorption at 3440 $\text{cm}^{-1}$  indicates the presence of water molecules. Complex (1) crystallizes in an orthorhombic space group  $\text{Pbc}a$ .

The symmetric unit of the title complex contains a dodeca-nuclear cube-octahedron  $[\text{Co}_{12}(\text{cpip})_{12}(\text{H}_2\text{O})_{12}]$ . This is shown as an ORTEP drawing along with the atom numbering scheme depicted in Figure 1 (a) and the representation of the cube-octahedron in Figure 1 (b). Also, a polyhedral model of the  $\text{Co}_{12}$  cube-octahedron is shown in Figure (2). Finally, the view of the packing diagram is shown in Figure 3. The carboxylate groups of the twelve  $\text{H}_2\text{cpip}$  ligands are bridging the twelve cobalt (II) ions, where each  $\text{cpip}^{2-}$  acts as a pentadentate ligand through four oxygen atoms of two carboxylate groups and one nitrogen atom (Scheme 2).

**Table 1.** Crystal data and structure refinement details of 1.

<b>Empirical formula</b>	<b>C<sub>120</sub> H<sub>96</sub> Co<sub>12</sub> N<sub>12</sub> O<sub>78.56</sub></b>
<b>Formula weight</b>	3670.14
<b>Temperature</b>	293(2) K
<b>Wavelength</b>	0.71073 Å
<b>Crystal system, space group</b>	Orthorhombic, P b c a
<b>Unit cell dimensions</b>	a = 31.3608(5) Å b = 31.4189(4) Å β = 90 deg. c = 31.7084(4) Å
<b>Volume</b>	31243.0(7) Å <sup>3</sup>
<b>Z, Calculated density</b>	8, 1.561 Mg/m <sup>3</sup>
<b>Absorption coefficient</b>	1.340 mm <sup>-1</sup>
<b>F (000)</b>	14820
<b>Crystal size</b>	0.205 x 0.188 x 0.072 mm
<b>Theta range for data collection</b>	2.951 to 25.100 deg.
<b>Limiting indices</b>	-37<=h<=37, -37<=k<=37, -37<=l<=37
<b>Reflections collected / unique</b>	53444 / 27752 [R(int) = 0.0910]
<b>Completeness to theta =</b>	25.100 99.8 %
<b>Refinement method</b>	Full-matrix least-squares on F <sup>2</sup>
<b>Data / restraints / parameters</b>	27752 / 0 / 1975
<b>Goodness-of-fit on F<sup>2</sup></b>	1.025
<b>Final R indices [I&gt;2sigma(I)]</b>	R1 = 0.0830, wR2 = 0.2300
<b>R indices (all data)</b>	R1 = 0.1819, wR2 = 0.2816
<b>Extinction coefficient</b>	n/a
<b>Largest diff. peak and hole</b>	1.666 and -0.752 e.Å <sup>-3</sup>

**Scheme 2**

Each cobalt (II) ion has a distorted octahedral coordination sphere and coordinates to two cpip<sup>2-</sup> ligands and one water molecule. Each ligand binds to one cobalt (II) ion through two oxygen atoms of two carboxylate groups and one nitrogen atom of the same ligand. At the same time, the rest of the coordination sites are occupied by another two oxygen atoms of two carboxylate groups of another cpip<sup>2-</sup> ligands and one terminal water molecule. All the oxygen atoms of the carboxylate groups of the ligand cpip<sup>2-</sup> occupied the equatorial planes, while the amine nitrogen atoms

of the cpip<sup>2-</sup> ligands and water molecules occupied the axial planes.

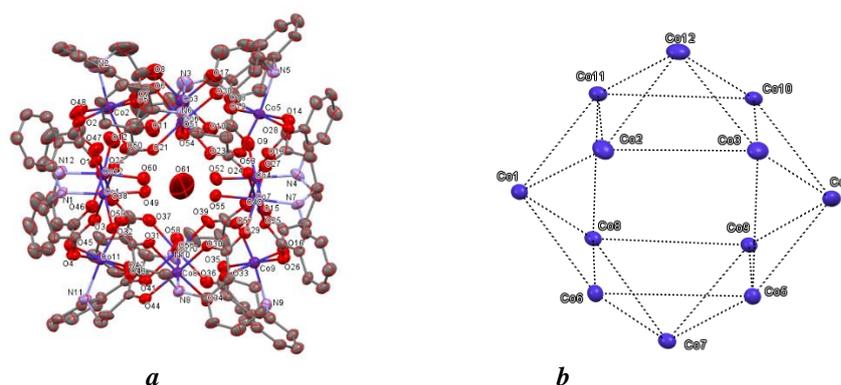
The carboxylic groups of all cpip<sup>2-</sup> ligands are deprotonated, and all exhibit one coordination mode within the aggregate; *syn(eq)-anti(eq)* bridging. The near equalities of the C-O bond distance of the carboxylate groups of the ligand cpip<sup>2-</sup>, C1/O1/O2 (1.24, 1.28 Å), C10/O3/O4 (1.26, 1.25 Å), C12/O5/O6 (1.24, 1.27 Å), C70/O27/O28, (1.26, 1.28 Å), C82/O33/O34 (1.28, 1.25 Å) indicate delocalized bonding arrangement rather than delocalized single and double bonds. Terminal water molecules attached

to the cobalt atoms are involved in H-bonding with the lattice water molecule that resides in the cavity of the metallocycle complex, ranging from 3.226 Å to

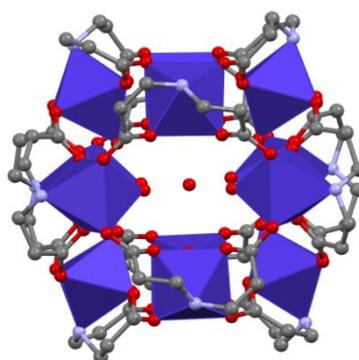
3.408 Å. There are H-bonding between the coordinated water molecules and the oxygen atoms of the acetate groups  $O_{w(49)}-H \cdots O(21) = 2.716$  Å,  $O(w)52-H \cdots O9 = 3.166$  Å and between the amine group and the oxygen of the carboxylate groups of the ligand  $N1-H \cdots O1 = 2.861$  Å. The structure contains disordered water molecules featuring H-bond contacts of  $O \cdots O$  type. Selected bond lengths and angles for complex (1) are given in (Table 2). The average Co-O bond length depends on the kind of ligand involved;

the Co-O (cpip2-) bonds are all in the range of 2.145 to 2.030 Å, while those involved coordinated terminal water molecules Co-O ( $H_2O$ ) bonds are in the range of 2.163 to 2.134 Å. All are following the previously reported compounds <sup>16</sup>.

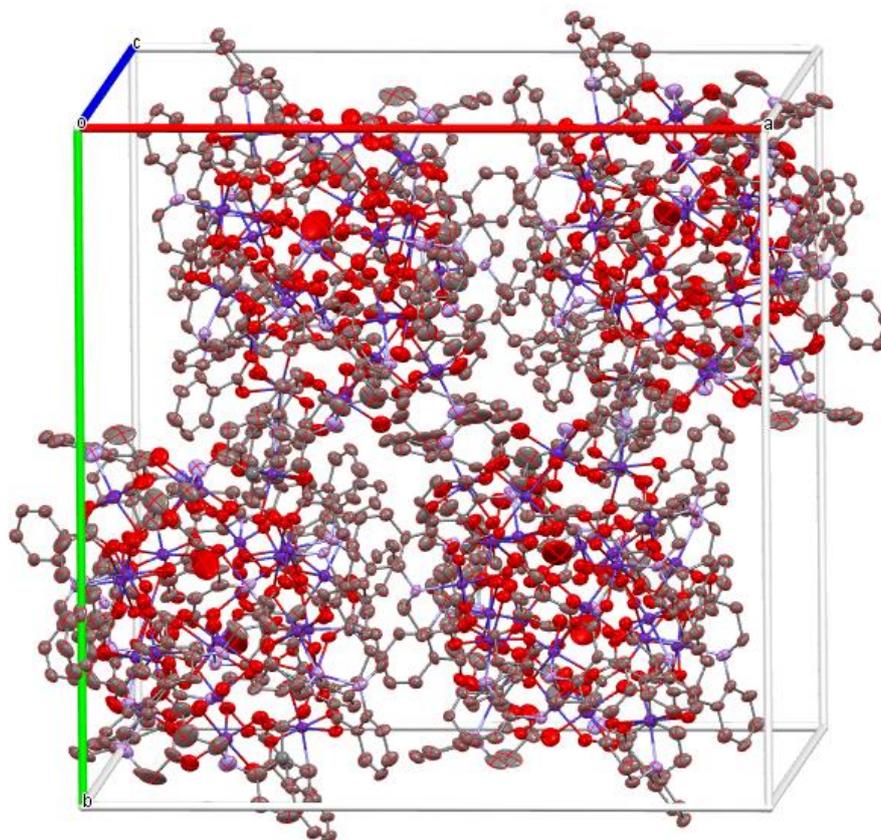
Intermetallic Co.....Co distances within the cube-octahedron are in the range of 7.52-5.18 Å, which is long compared with (3.17 Å to 3.12 Å) in  $[Co_{12}(chp)_{12}(OCMe)_{12}(H_2O)_{12}(THF)_6]$  (chp = 6-chloro-2-pyridonate) <sup>17</sup> and (3.22 to 3.18 Å) in  $[Co_{12}(\mu-O)_3(\mu 1,1,1-N_3)_3(\mu_3-L)_9(\mu-L)_6](ClO_4)_2 \cdot H_3tea \cdot 9.5H_2O$  where  $L = 1$  H-benzimidazol-2-YL) methanol and  $H_3tea =$  tri-ethanol amine <sup>8</sup>.



**Figure 1. a.** Single crystal structure of the title complex (1) with 50% probability thermal ellipsoids showing the atom labeling scheme. Hydrogen and the rest of the ligand atoms are omitted for clarity. **b.** Representation of cube-octahedron



**Figure 2.** A polyhedral representation of  $Co_{12}$  cube-octahedron



**Figure 3.** Packing diagram of complex (1)

**Table 2.** Selected bond lengths [ $\text{\AA}$ ] and angles [deg] for complex (1).

BondLengths( $\text{\AA}$ )

<b>Co(1)-O(1)</b>	<b>2.025(7)</b>	<b>Co(1)-O(32)</b>	<b>2.082(6)</b>	<b>Co(1)-O(3)</b>	<b>2.092(7)</b>
<b>Co(1)-N(1)</b>	2.136(8)	Co(1)-O(22)	2.145(7)	Co(1)-O(49)	2.151(7)
<b>Co(2)-O(2)</b>	2.048(8)	Co(2)-O(5)	2.055(7)	Co(2)-O(7)	2.066(9)
<b>Co(2)-O(48)</b>	2.086(9)	Co(2)-O(50)	2.157(7)	Co(2)-N(2)	2.174(10)
<b>Co(3)-O(10)</b>	2.040(8)	Co(3)-O(11)	2.048(8)	Co(3)-O(17)	2.065(7)
<b>Co(3)-O(8)</b>	2.094(10)	Co(3)-N(3)	2.142(10)	Co(3)-O(51)	2.150(7)
<b>Co(4)-O(13)</b>	2.062(7)	Co(4)-O(15)	2.068(7)	Co(4)-O(40)	2.100(8)
<b>Co(4)-O(9)</b>	2.128(8)	Co(4)-O(52)	2.135(7)	Co(4)-N(4)	2.140(9)
<b>Co(5)-O(18)</b>	2.029(7)	Co(5)-O(28)	2.071(7)	Co(5)-O(19)	2.101(7)
<b>Co(5)-O(14)</b>	2.132(7)	Co(5)-O(53)	2.141(7)	Co(5)-N(5)	2.147(8)
<b>Co(6)-O(21)</b>	2.051(7)	Co(6)-O(23)	2.071(7)	Co(6)-O(20)	2.105(7)
<b>Co(6)-O(6)</b>	2.130(7)	Co(6)-N(6)	2.137(8)	Co(6)-O(54)	2.145(7)

Bond Angles ( $^{\circ}$ )

<b>O(1)-Co(1)-O(32)</b>	<b>177.8(3)</b>	<b>O(1)-Co(1)-O(3)</b>	<b>88.0(3)</b>
<b>O(32)-Co(1)-O(3)</b>	90.0(3)	O(1)-Co(1)-N(1)	86.8(3)
<b>O(32)-Co(1)-N(1)</b>	92.5(3)	O(3)-Co(1)-N(1)	90.8(3)
<b>O(1)-Co(1)-O(22)</b>	92.2(3)	O(32)-Co(1)-O(22)	89.8(3)
<b>O(3)-Co(1)-O(22)</b>	179.5(3)	N(1)-Co(1)-O(22)	88.7(3)
<b>O(1)-Co(1)-O(49)</b>	84.9(3)	O(32)-Co(1)-O(49)	95.8(3)

#### 4. Conclusion

We presented the synthesis and crystal structure analysis of the dodecahedron cobalt (II) complex. The  $\text{cpip}^{2-}$  acts as a pentadentate ligand in its coordination with cobalt (II) centers through four oxygen atoms of two carboxylate groups and one nitrogen atom. Hydrogen bonding between a lattice water molecule and coordinated water molecules adds more stability to the complex. The magnetic properties of this complex are of high interest due to the presence of twelve cobalt atoms of oxidation state +2, making it a high candidate to be a single magnetic molecule (SMMs).

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